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File: USPT

DOCUMENT-IDENTIFIER: US H000521 H

TITLE: Thermosetting polysulfones

Abstract Text (2):

wherein Z and Z' are each a monovalent unsaturated organo radical. Usually Z and Z' are alkylene, aralkylene or cycloalkylene moieties. The end-capped polyarylene polyethers can be cured as is or in the presence of one or more unsaturated comonomers to afford homopolymers or copolymers, respectively. Such cured systems exhibit high glass transition temperatures, good tensile properties, excellent electric and alkali resistance and improved stress cracking resistance. End-terminated polysulfone resins having molecular weight of 5,000 to 15,000 are especially advantageous. The properties exhibited by the vinyl/allyl terminated oligomers are useful in fields which require high temperature performance, excellent solvent resistance and good fabrication characteristics. Specific areas of application include high performance molded products for appliances and electronics, high temperature laminates and adhesives and protective and insulative coatings.

Brief Summary Text (23):

The thermoset vinyl benzyloxy end-capped polysulfones in coating form on metal sheets and the like are highly resistant to the solvent action to acetone and chlorinated hydrocarbons, such as, methylene chloride. Such coatings are also highly resistant to hot alkali solutions.

Brief Summary Text (25):

The thermoset vinylbenzyloxy end-capped polysulfone (10,000 molecular weight) had significant resistance to the solvent action of trichloroethylene, which is important in some electrical applications.

Detailed Description Text (6):

Dihydric phenol, such as, 2,2-bis(4-hydroxyphenyl)propane ("bisphenol-A"), is dissolved in a solvent such as a mixture of monochlorobenzene and dimethyl sulfoxide. The dihydric phenol is converted to the alkali metal salt by adding an alkali metal hydroxide, such as, sodium hydroxide, an alkali metal hydride, an alkali metal hydroxide, an alkali metal alkoxide or an alkali metal alkyl compound, and removing the water of condensation by azeotropic distillation. An aromatic compound having two activated halo substituents is added to the alkali metal salt. 4,4'-Dichlorodiphenyl sulfone is illustrative of such aromatic compounds. The dihalo aromatic compound is used in a controlled proportion so that there will be a stoichiometric excess of the alkali metal salt. The dihalo aromatic compound and the alkali metal salt are reacted to form a linear polyarylene-polyether chain having alkali metal salt end groups. This compound is then reacted with an appropriate end-capping agent, such as, allyl chloride, to form a monovalent unsaturated organo end-capped polyarylene-polyether, which is recovered by coagulating the solution in anhydrous methanol or anhydrous isopropyl alcohol.

Detailed Description Text (13):

In the first step, that is, reaction step (1), the dihydric phenol is converted to the corresponding alkali metal salt. Two moles of alkali metal hydroxide, such as sodium hydroxide or potassium hydroxide, are reacted per mole of dihydric phenol. Almost exactly stoichiometric quantities should be used. This reaction is carried out in a solvent system that permits azeotropic removal of the water of condensation. A mixture of monochlorobenzene (MCB) and dimethyl sulfoxide (DMSO) is excellent for this purpose. The DMSO is used as the solvent, and MCB is an azeotroping agent. Other useful solvents include dimethylacetamide (DMAC), and other useful azeotroping agents include chlorinated benzenes, benzene, toluene, and xylene. The condensation reaction to

produce the alkali metal phenoxide normally is conducted from about 120 to about 240 minutes at a temperature of from about 110.degree. to about 132.degree. C. While a much broader temperature range can be used, the above one is the most convenient.

Detailed Description Text (20):

The end-capping reaction, i.e., reaction step (3), is carried out by reacting the monovalent unsubstituted organo compound with the alkali metal phenoxide-capped polyarylene polyether resin produced by reaction step (2). The stoichiometric proportions are two moles of unsubstituted organo compound per mole of polyarylene polyether resin. It is preferred to employ about a 2 to 10 mole percent stoichiometric excess of the unsubstituted organo compound. The reaction mixture preferably should be substantially anhydrous. The reaction medium can be the same solvent system that was employed for reaction steps (1) and (2). The reaction is carried out at elevated temperatures, e.g., at from about 110.degree. C. to about 165.degree. C. At the recommended temperature range, the reaction will usually take from about 10 to about 90 minutes. The completion of the reaction can be detected by treating a sample of the reaction mixture with bromocresol purple indicator. When the alkali metal phenoxide has reacted, the treated sample will be greenish yellow in color.

Detailed Description Text (21):

At the completion of the reaction, the reaction mixture can be cooled, filtered to remove salt by-product, and then the end-capped polyarylene polyether resin can be recovered by coagulation in a non-solvent for the resin, e.g., methanol or isopropyl alcohol. Preferably the reaction mixture and the resin is kept anhydrous until the resin has been recovered as a solid.

Detailed Description Text (22):

The substantially equimolar one-step reaction of a double alkali metal salt of a dihydric phenol with a dihalobenzenoid compound is done in the presence, for example, of specific liquid organic sulfoxide or sulfone solvents under substantially anhydrous conditions. Catalysts are not necessary for this reaction. Another feature of the invention involves simultaneously contacting substantially equimolar amounts of an alkali metal double salt of a dihydric phenol and a dihalobenzenoid compound with a solvent mixture comprising an azeotrope former and a sulfoxide or sulfone reaction solvent in a weight ratio of from about 10:1 to about 1:1, preferably from about 4:1 to about 3:1, removing water from the reaction mass as an azeotrope with the azeotrope former until substantially anhydrous conditions are attained, adjusting the ratio of azeotrope former to reaction solvent from about 1:1 to about 1:10, preferably from about 1:3 to about 1:4, by removing excess azeotrope former, and reacting the alkali metal double salt with the dihalobenzenoid compound in the liquid phase of the sulfoxide or sulfone reaction solvent.

Detailed Description Text (23):

The useful sulfoxide or sulfone solvents are those of the formula:

Detailed Description Text (24):

wherein each R represents a monovalent lower hydrocarbon group free of aliphatic unsaturation of the alpha carbon atom, and preferably contains less than about 8 carbon atoms or when connected together represents a divalent alkylene group with v being an integer from 1 to 2 inclusive. Thus, in all of these solvents all oxygens and two carbon atoms are bonded directly to the sulfur atom. Thus, contemplated for use in the invention are such solvents as those having the formula: ##STR7## wherein the R groups are lower alkyl, such as methyl, ethyl, propyl, butyl and like groups and aryl groups such as phenyl and alkyl phenyl groups as well as those where the R groups are interconnected as in a divalent alkylene bridge, such as: ##STR8## as in thiophene oxides and dioxides. Specifically mentionable of these solvents but by no means exhaustive of these solvents are dimethylsulfoxide, dimethylsulfone, diethylsulfoxide, diethylsulfone, diisopropylsulfone, tetrahydrothiophene 1,1-dioxide (commonly called tetramethylene sulfone or sulfolane) and tetrahydrothiophene-1 monooxide. The dimethylsulfoxide has been found to be the most useful as the solvent for this reaction because it is a solvent for the widest variety of reactants as well as for the resulting polymer of the reaction. Aliphatic unsaturation on the alpha-carbon atom, such as occurs in divinyl sulfone and sulfoxide, should not be present as such materials tend to be reactive and polymerize under the conditions of this reaction. However, unsaturation on a beta-carbon atom or one further removed from the sulfur atom can be tolerated and such solvents can be employed in this reaction.

Detailed Description Text (25):

In situations where it is desired to prepare the alkali metal salt of the dihydric

phenol in situ in the reaction solvent, the dihydric phenol and an alkali metal hydroxide are admixed in essentially stoichiometric amounts and normal precautions taken to remove all the water of neutralization preferably by distillation of a water-containing azeotrope from the solvent-metal salt mixture. It has been convenient to employ benzene, xylene, halogenated benzenes or other inert organic azeotrope-forming organic liquids in performing this. Heating the alkali metal hydroxide, dihydric phenol and small amounts of the azeotrope former to reflux for several hours while removing the azeotrope is the most desirable. However, it is obvious that any other technique for removing essentially all of the water can be equally satisfactory.

Detailed Description Text (26):

It is not essential and critical in this reaction that all of the azeotropic former be removed before the reaction of the alkali metal salt of the bisphenol with the dihalobenzenoid compound. In fact, it is desirable in some instances to employ an amount of such material in excess of that needed to azeotrope off all of the water, with the balance being used as a co-solvent or inert diluent with the sulfone or sulfoxide principal solvent. Thus, for instance, benzene, heptane, xylene, toluene, chlorobenzene, dichlorobenzene or like inert liquids can be beneficially employed.

Detailed Description Text (27):

The azeotrope former can be one either miscible or immiscible with the sulfone or sulfoxide major solvent. If it is not miscible it should be one which will not cause precipitation of the polymer in the reaction mass. Heptane is such a solvent. When employed, it will merely remain inert and immiscible in the reaction mass. If the azeotrope former would cause precipitation of the polymer, it should be removed almost completely from the reaction mass before initiating polymerization. For such reasons, it is preferred to employ azeotrope formers which are miscible with the major solvents and which also act as co-solvents for polymer during polymerization. Chlorobenzene, dichlorobenzene and xylene are azeotrope formers of this class. Preferably the azeotrope former should be one boiling below the decomposition temperature of the major solvent and be perfectly stable and inert in the process, particularly inert to the alkali metal hydroxide when the alkali metal salt of the dihydric phenol is prepared in situ in the presence of the inert diluent or azeotrope former. It has been found that chlorobenzene and o-dichlorobenzene serve particularly well as the inert diluent and are able to significantly reduce the amount of the sulfone or sulfoxide solvent necessary. The co-solvent mixture using even as much as 50 percent of the halogenated benzene with dimethylsulfoxide, for example, not only permits the formed polymer to remain in solution and thus produce high molecular weight polymers, but also provides a very economical processing system, and an effective dehydration operation.

Detailed Description Text (29):

Substantially equimolar amounts of the alkali metal double salt of a dihydric phenol (or a dihydric phenol for in situ conversion to the salt) and a dihalobenzenoid compound are simultaneously contacted with a solvent mixture comprising an azeotrope former and a sulfoxide or sulfone reaction solvent in a ratio of from about 10:1 to about 1:1 by weight based on the combined weight of the azeotrope former and reaction solvent. When water is present in this solvent mixture, a phase separation occurs even between normally miscible liquids creating two liquid phases. When this phase separation occurs, the water and hydrated alkali metal double salt of a dihydric phenol are preferentially dissolved in the sulfoxide or sulfone reaction solvent phase and the dihalobenzenoid compound is preferentially dissolved in the azeotrope former phase. The initial ratio of azeotrope former to reaction solvent is critical in this respect because with lesser amounts of azeotrope former, two liquid phases are not formed and undesirable hydrolysis of the dihalobenzenoid compound occurs. Only when the ratios specified are used does a phase separation occur which isolates the dihalobenzenoid compound from hydrolysis by the water in the system.

Detailed Description Text (30):

If dry crystalline alkali metal salt of a dihydric phenol is employed, problems relative to obtaining anhydrous conditions are essentially avoided. However, drying the salt and keeping it dry during transfer and charging is difficult. It is more advantageous to employ a hydrated alkali metal salt which is dehydrated in the reaction mass by removing the water of hydration. Water then can be present or formed in the reaction mass as the water of hydration of a hydrated alkali metal salt, as the water of neutralization formed during the in situ conversion of a dihydric phenol to the alkali metal double salt, or as water present in either the azeotrope former or the reaction solvent. During the in situ conversion, the hydrated salt is formed first which is then dehydrated upon the removal of water.

Detailed Description Text (31):

When the two liquid phases are formed, it is important that the water in the system be removed as quickly as possible, usually by refluxing the mixture at about the boiling point of the azeotrope former until substantially all of the water is removed. During the water removal, the dehydrated alkali metal double salt, which is insoluble in the reaction solvent at the reflux temperatures, precipitates. The fact that all the water has been removed is usually signaled by no further precipitate formation, no further azeotrope formation, and the formation of one liquid phase when miscible liquids are used. After the water has been removed, excess azeotrope former is removed by distillation until the ratio of azeotrope former to sulfoxide or sulfone reaction solvent is about 1:1 to 1:10. Only when the amount of azeotrope former is reduced to within these ratios, does significant polymerization occur.

Detailed Description Text (44):

Dimethylsulfoxide decomposes at its boiling point, i.e., about 189.degree. C.; it is desirable to keep the reaction temperature below this to avoid such problems when employing this solvent.

Detailed Description Text (45):

The reaction temperature can be effectively increased even above the normal boiling point of the solvent or mixture of solvents by the use of pressure in the system. However, for most practical reactions contemplated herein, atmospheric pressures are quite adequate, though if desired pressures as high as 1000 psig or more can be employed.

Detailed Description Text (46):

The molecular weight of the polymer can be easily controlled in this process by the addition of a precipitating solvent to the reaction mixture when the desired reduced viscosity of the resin is secured or when the indicated viscosity of the polymerization mass is high enough to indicate the desired molecular weights are achieved. It is also possible to terminate the growing polymer chain by the addition of a monofunctional chain stopper, such as an alkyl halide or other suitable coreactant.

Detailed Description Text (47):

The molecular weight of the end-capped resins is indicated by reduced viscosity in indicated solvents. The viscosity of a resin solution bears a direct relationship to the weight average molecular size of the resin chains, and can be used to characterize the degree of polymerization. The reduced viscosity values used herein are of significance only relative to each other rather than in any absolute sense, and for this reason other polyether-solvent systems can be employed as an indication of the relative molecular weight of these resins. When solvents other than chloroform are employed, the required average molecular size relationship can readily be established by reference to the reduced viscosity values defined herein even though the numerical reduced viscosity values of the alternative system may be different.

Detailed Description Text (48):

In the in situ process (conversion) the preferred solvent is dimethylacetamide; the process uses K.sub.2 CO.sub.3.

Detailed Description Text (56):

The polymerization step is carried out at a temperature preferably between 163.degree. and 165.degree. C. in a mixed solvent composed of dimethyl sulfoxide (DMSO) and monochlorobenzene (MCB), although any other suitable solvent(s) such as dimethyl acetamide (DMAC) can also be employed. Any suitable polymerization temperature (e.g., 120.degree. to 185.degree. C.) can be used. Precautions which are normally observed during the preparation of standard polysulfone are equally important here to achieve quality products. Of particular importance is the caustic charge which affects both the "color" and "stability" of the finished polysulfone-vinyl reactive resin in addition to its anticipated effect on the molecular weight and composition of the latter.

Detailed Description Text (57):

Useful polar solvents include: alcohols, such as, methanol, ethanol, n-propyl alcohol, n-butyl alcohol, amyl alcohol, isopropyl alcohol, sec-butyl alcohol, isobutyl alcohol and 4-methyl 2-pentanol; ethers, such as ethyl ether, diethyl Cellosolve and butyl ether; aldehydes, such as, benzaldehyde and furfural; triethyl phosphate; and amides, such as, N,N-dimethyl formamide, acetamide and butramide.

Detailed Description Text (63):

Preferably anhydrous condition during the end-capping stage, coupled with a reasonably good hydrolytic stability of the halo organo compound, is used whereby premature gelation of the polysulfone-vinyl reactive resin is normally not a problem in the reactor. Upon completion of the end-capping step, the reaction mixture should be neutral which can be readily diluted with dry monochlorobenzene (or other suitable solvent) and filtered without difficulty. However, the presence of residual alkalinity, which may result from either a deficiency in end-capping reagent charged or an incompleteness in reaction, can cause problems during subsequent filtering when the polymer solution is exposed to either atmospheric moisture and/or wet diluent. For this reason a speedy product recovery is recommended whenever the above circumstance is present or suspected.

Detailed Description Text (65):

The polysulfone-vinyl reactive resin can be separated or recovered from the reaction mixture by any appropriate method, but the coagulation method and the melt recovery process are preferred. Recovery by coagulation is normally done in isopropanol (or methanol). The melt recovery production process involves removing the solvents by passing the polymer solution through a recovery system consisting of a concentrator, a devolatilizer and a Marshall mill.

Detailed Description Text (66):

While polysulfone-silane reactive resins are outside of the scope of the invention, they are frequently used herein for comparative purposes. The polysulfone-silane reactive resins are structurally very similar to the polysulfone-vinyl reactive resins of the invention, except for the end-capping by reaction with silane groups such as 3-chloropropyltrimethoxysilane. The production process is very similar for the two types of end-capped polysulfones. The polysulfone-silane reactive resins have been prepared in a broad range of molecular weights by varying the monomer feed ratio in the synthesis of the oligomers. The polysulfone-silane reactive resins have excellent hydrolytic stability in bulk form below their glass transition temperature. They can even be suspended in water without premature gelation. Cross-linking occurs readily above the glass transition temperature, and the cure rate can be further increased by prehydrolysis of the silylalkoxy end groups or by the use of catalysts. Cross-linked polysulfone-silane reactive resins have mechanical and electrical properties comparable to those of standard polysulfone. Cured polysulfone-silane reactive may enable higher use temperature limits than thermoplastic polysulfone resins, and large improvements over such thermoplastic polysulfone resins are found in their solvent resistance (environmental stress crack resistance). Also, due to the silane end groups, a built-in bonding capability exists towards many inorganic and metallic surfaces.

Detailed Description Text (68):

Due to the absence of moisture sensitive end groups, the polysulfone-vinyl reactive resins are usually easier to handle than polysulfone-silane reactive resins during preparation. Once the end-capping reaction is complete, the polysulfone-vinyl reactive resins may be exposed to water or wet solvent without being affected. The only exceptions, however, are the polysulfone-vinyl reactive resins having methacryloxy or acryloxy end groups which are hydrolyzed readily by moisture. For this reason, the chloroalkyl esters of such are preferred.

Detailed Description Text (83):

Polysulfone has exceptional chemical resistance, particularly in a strong alkaline environment. This quality makes the polysulfone-vinyl reactive resin a suitable matrix resin for fabricating corrosion-resistant composite materials. Polysulfone-vinyl reactive resins, while retaining most of the polysulfone's properties, exhibit a number of additional features which are highly desirable for the coating applications. Because of their oligomeric nature, they are characterized by high solubilities in polysulfone-soluble solvents, low melting points and good melt flow characteristics which make them suitable for a variety of fabrication processes, particularly the solution and power coating techniques. The reactive end-groups of the polysulfone-vinyl reactive resins permit curing by either heat, peroxide, U.V., electron-beam radiation or other suitable energy sources. Once cured, the coating is transformed to a clear, rigid, tough thermoset possessing excellent solvent, chemical and thermal resistance. In contrast to the silane end-capped counterparts, polysulfone-vinyl reactive resins lack inherent bonding capabilities. Better adhesion, however, can be achieved by modifying with say unsaturated silane or silyl peroxide adhesion promoters or suitable polar comonomers. The polysulfone-vinyl reactive resins can be for a variety of coating applications including magnet wire coatings, photocurable coatings and powder coatings.

Detailed Description Text (85):

The polysulfone-vinyl reactive resins are also useful as modifiers for other (reactive) polymers to impart certain desirable polysulfone properties. The resulting compositions can be either copolymers or interpenetrating polymeric networks (IPN's). In addition, the polysulfone-vinyl reactive resins can be used for formulating photocurable printing plates and for making membranes to take advantage of their mechanical rigidity, and chemical resistance; porosity in the membrane can be readily developed by solvent leaching upon curing.

Detailed Description Text (88):

End-capping of 5,000 molecular weight, hydroxy terminated polysulfone oligomer with vinylbenzyl chloride afforded a thermosetting composition with excellent solvent resistance. At 200 psi, this material did not rupture after twenty-five minutes of exposure of acetone, toluene, and trichloroethylene. Although compression molded plaques were brittle the high cure speed and good flow behavior should make this material of interest in coating applications (e.g., powder coating).

Detailed Description Text (89):

The properties of the thermosetting polysulfone-vinyl reactive resin and their blends with some epoxy resins are listed in Table A below: The 5,000 MW polysulfone-vinyl reactive resin/epoxy system exhibited excellent flow characteristics, solvent resistance, a high glass transition temperature (Tg) and good tensile properties.

Detailed Description Text (104):

The co- or homopolymerization can be effected using slurry suspension or emulsion type polymerization (with conventional aqueous and non-aqueous solvents and liquid carriers).

Detailed Description Text (129):

Compounding--The peroxide, usually DiCup, was dissolved in the liquid comonomer, and was subsequently mixed with the polysulfone-vinyl reactive resin fluff. When the two components were immiscible, a common solvent of low boiling point, such as acetone, was used to give a homogeneous solution before blending with the resin fluff. The solvent was removed by air drying. The latter method usually provides a more uniform mixture, but some comonomer may be lost due to evaporation.

Detailed Description Text (138):

The gel contents of the cured polysulfone-vinyl reactive resin samples were measured by solvent extraction in boiling chloroform. The data of percent extractables shown in Tables XVI and XVIII were determined in methylene chloride. The higher the cross-linking is, the greater the gel content and the lower the percent extractables.

Detailed Description Text (142):

Environmental stress-crack resistance data were measured at constant stress levels using an environment of solvent-saturated cotton swab attached to the specimen. Performance was rated on the basis of time elapsed before rupture at a given stress level.

Detailed Description Text (159):

Allylic terminated polysulfone prepared as in Example 1 above was dissolved in methylene chloride solvent to provide a 10 percent solids-containing solution. Several steel "D" plates, 10 mils thick, were dip coated in this solution and dried at room temperature for 10 minutes which was followed first by, oven drying at 125.degree. C. for 45 minutes and then by various high temperature oven time bake exposures at 315.degree. to 320.degree. C.

Detailed Description Text (160):

Upon weighing the residual coating baked onto the steel, the coatings were subjected to CH.sub.2 Cl.sub.2 extraction by immersion in the stirred solvent bath. The appropriate graph in FIG. 1 illustrates that the degree of solubility to methylene chloride is an inverse function of bake time exposure in an oven at 315.degree. to 320.degree. C. Similar extraction data following the identical procedure outlined above was obtained for glass and chrome substrates coated allylic terminated polysulfone (prepared as in Example 1 above) and for controls which had P-1700 polysulfone coatings. The appropriate graph in FIG. 1 illustrates the methylene chloride solubility curve for the glass-plated substrates; and the graph in FIG. 2 illustrates the methylene chloride solubility curve for the chrome-plated substrates. Steel "D" plates coated with either P-1700 polysulfone coatings or the allyl-terminated polysulfone (prepared as in Example 1) were subjected to acetone and methylene chloride extraction by immersion. The data

and results are set out below in Table II:

Detailed Description Text (161):

The data of Table II shows that acetone and methylene chloride immersion caused stress cracking or dissolution of the P-1700 polysulfone coatings, but did not have any effect on the allyl-terminated polysulfone coatings of this invention. Several samples of aluminum sheet (7.5 mils thick, type 3003 H 14) were also coated as described in the procedure above and baked. The coated aluminum plates were subjected to acetone and methylene chloride extraction by immersion, in some cases followed by dipping in hot sodium hydroxide solution. A comparison of the solvent resistance (data and results) of allyl terminated polysulfone and conventional P-1700 polysulfone is set out below in Table III:

Detailed Description Text (162):

The data of Table III shows that acetone and methylene chloride immersion caused stress cracking or coating removal of the P-1700 polysulfone coatings, but did not have any effect on the allyl-terminated polysulfone coatings of this invention. Dipping in hot sodium hydroxide solution, after the solvent immersion step, had no effect with the allyl-terminated polysulfone coatings but did attach the aluminum substrates associated with the P-1700 polysulfone coatings.

Detailed Description Text (163):

Both the allylic-terminated polysulfone and P-1700 polysulfone coatings released from glass substrates after baking when exposed to hot detergent solutions (80.degree. C.--14 minutes). On one case, the control P-1700 did not show chlorinated solvent resistance whereas the allyl terminated and baked coating provides solvent resistance (chlorinated and acetone) yet may be readily removed with hot detergent solutions.

Detailed Description Text (180):

The 0.22 RV, 2-chloroethyl methacrylate end-capped polysulfone-vinyl reactive resin shown in Table IV is an example of low end-capping efficiency. Its deficiency is reflected by the higher than expected number average molecular weight (M.sub.n) value calculated on the assumption of 100 percent end-capping efficiency. Such a resin may contain up to 30 percent of non-curable fraction as evidenced by solvent extraction test. When the above mentioned precautions are observed during the preparation, however, this difficulty may be largely alleviated.

Detailed Description Text (203):

Upon cross-linking, polysulfone-vinyl reactive resins exhibit a marked improvement in environmental stress-crack resistance over the regular polysulfone, similar to that of the polysulfone-silane reactive resins. The degree of improvement is usually parallel to the increase in cross-linking density of the cured material. This effect is demonstrated clearly by the data shown in Table XIII below where the environmental stress crack resistance of a series of cured polysulfone-vinyl reactive styrene resins are related to their swelling indices. The cross-linking density is expected to increase with a decrease in the initial molecular weight of the polysulfone-vinyl reactive, which is reflected by a lowering in the swelling index. The environmental stress-crack resistance data was measured at constant stress levels using an environment of solvent-saturated cotton swab attached to the specimen.

Detailed Description Text (205):

The environmental stress-crack resistance of a cured polysulfone-vinyl reactive comonomer composition also depended on the type and loading of comonomer employed. (The hydroxy terminated polysulfones which were vinyl end-capped were prepared from bisphenol A and 4,4 'dichlorodiphenyl sulfone as described in Example 1.) Table XIV below sets out the environmental stress-crack resistance performance, in decreasing order, of 20 different types of matrices in toluene and trichloroethylene. Three of the more effective comonomers among those studied were divinyl benzene, acrylonitrile and styrene. The neat polysulfone-vinyl reactive resin also performed well upon cross-linking and is comparable to cross-linked polysulfone-silane reactive resin. When styrene was the comonomer used, the resistance of the cured material to solvent-induced stress-crack deteriorated with increasing comonomer loadings.

Detailed Description Text (207):

The solution copolymerization of a polysulfone-vinyl reactive resin (acryloxy ethoxy end-groups, 0.23 initial R. V.) with styrene in mono-chlorobenzene was carried out to partial competition to minimize the tendency of premature crosslinking. Nevertheless, when the weight percent of PSF-VR in the feed exceeded 30 percent, complete gelation usually resulted. Between 12.5 and 30 percent polysulfone-vinyl reactive loadings in

the feed provided partially gelled products. Soluble copolymers were also prepared at polysulfone-vinyl reactive charges below 12.5 percent. The characteristics of some of the soluble copolymers are shown in Table XV below. Compositions of these copolymers were determined by elemental analysis. In every instance the polysulfone-vinyl reactive was found to be richer in the copolymer than in the feed. They are soluble in methyl ethyl ketone or toluene, which are non-solvents for the polysulfone-vinyl reactive homopolymers. The molecular weights of the products increased rapidly with increasing polysulfone-vinyl reactive/styrene charge ratios. These thermoplastic resins can be compression-molded to clear or slightly hazy plaques; they are usually brittle and resemble polystyrene.

Detailed Description Text (236):

Into a 1-liter, four-necked flask, equipped with a mechanical stirrer, water trap, addition funnel, thermometer and argon inlet, there was placed 101.12 grams (0.4 mole) of 4,4'-sulfonyl-diphenol, 510 milliliters of monochlorobenzene and 250 milliliters of DMSO. Upon dissolution at 75.degree. C., a caustic solution containing 16.24 grams (98.5 percent purity, 0.8 mole) of sodium hydroxide and 37.5 milliliters of distilled water was introduced. Azeotropic distillation was carried out until the temperature of the reaction medium reached 155.degree. C. when a total of 290.4 grams of distillate was collected. The reaction temperature was lowered to 140.degree. C. and a hot solution of 97.55 grams (0.34 mole) of sulfone monomer in 12 milliliters of dry monochlorobenzene was added. An additional 441.8 grams of solvent was distilled off to raise the reaction temperature to 165.degree. C. It was kept at this temperature for 3 hours. The polymer had the following structural formula: ##STR25## The polymer was end-capped with vinyl benzyl groups via the sodium phenolate end groups following the procedure of Example 2 using vinyl benzyl chloride.

Detailed Description Text (262):

To a solution of 24.26 g of 4,4'-dihydroxy diphenyl ether (0.12 mole) in 160 cc. of dimethyl sulfoxide and 45 cc. benzene in a 500 cc. reaction flask was added under a nitrogen atmosphere 25.27 g of 53.28 percent KOH (0.24 mole). The mixture was refluxed with stirring and slow nitrogen sparge with removal of water by a Dean-Stark trap for a total of 5 hrs. The mixture of solid potassium salt of 4,4'-dihydroxydiphenyl ether and solvent was cooled to room temperature and 34.46 g of 4,4'-dichlorodiphenylsulfone (0.12 mole) added. The reaction mixture was then warmed to about 130.degree. C. for 3.5 hours during which time it became quite viscous. The mixture was cooled to 110.degree. C. to 120.degree. C. and methyl chloride bubbled in for a short time to methylate any unreacted phenoxide groups. The polymer had the following structural formula: ##STR34## The polymer was end-capped with allyl groups via the potassium phenolate end groups following the procedure of Example 1 using allyl chloride.

Detailed Description Paragraph Table (5):

TABLE II	POLY- SOLVENT SULFONE AND SUBSTRATE
TYPE TREATMENT RESULTS	Steel "D" P-1700 Acetone, Stress cracked Plate 1 minute coating. immersion. Allyl Acetone, No effect. Terminated 1 minute immersion. P-1700 Methylene Coating dissolved. Chloride, 10 minute immersion. Allyl Methylene No effect. Terminated Chloride, 10 minute immersion. P-1700 Acetone, Coating dissolved. 10 minute immersion. Allyl Acetone, No effect. Terminated 10 minute immersion.

Detailed Description Paragraph Table (6):

TABLE III	POLY- SOLVENT SUB- SULFONE AND
STRATE.sup.1 TYPE TREATMENT RESULTS	Aluminum
P-1700 1 minute in Aluminum attacked. Acetone followed by 15 sec. dip in 20% NaOH solution. Allyl 1 minute in No effect. Terminated Acetone followed by 15 sec. dip in 20% NaOH solution. P-1700 10 minutes in Coating removed; CH.sub.2 Cl.sub.2 all metal attacked. followed by hot NaOH solution. Allyl 10 minutes in No effect. Terminated CH.sub.2 Cl.sub.2 followed by hot NaOH solution. P-1700 10 minute Stress cracked immersion coating. in Acetone. Allyl 10 minute No effect. Terminated immersion in Acetone.	Note: .sup.1 All substrates were given at least one 90.degree. bend in the test coupon prior to <u>solvent</u> resistance testing.

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L5: Entry 1 of 2

File: USPT

US-PAT-NO: 6026968

DOCUMENT-IDENTIFIER: US 6026968 A

TITLE: Reverse osmosis composite membrane

DATE-ISSUED: February 22, 2000

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Hachisuka; Hisao	Osaka			JP
Ikeda; Kenichi	Osaka			JP

US-CL-CURRENT: 210/490; 210/500.21, 210/500.27, 210/500.38, 427/245

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KMC
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☐ 2. Document ID: US 4780205 A

L5: Entry 2 of 2

File: USPT

US-PAT-NO: 4780205

DOCUMENT-IDENTIFIER: US 4780205 A

TITLE: Permselective hollow fiber membrane, process for the preparation thereof, method and apparatus for plasma components separation

DATE-ISSUED: October 25, 1988

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Murakami; Eiichi	Yamaguchi			JP
Matsuzawa; Kimihiko	Yamaguchi			JP
Masunaga; Eiji	Yamaguchi			JP

US-CL-CURRENT: 210/321.8; 210/500.23

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KMC
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